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Integrated Data Collection Analysis (IDCA) Program--FY2011 Project Descriptions

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Integrated Data Collection Analysis (IDCA) Program FY2011—Project Descriptions

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ABSTRACT

This document provides brief descriptions of research topics for consideration by the IDCA for potential funding in funding in FY 2011. The topics include the utilization of the results from the Proficiency Test developed during FY 2010 to start populating the small-scale safety and thermal testing (SSST) Testing Compendium and revising results from methods modifications. Other research topics were also developed for FY 2011 from issues that arose in the Proficiency Test.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX.

1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory small-scale safety and thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives. The materials for the Proficiency Test have been selected to span the challenging experimental issues arising when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

The IDCA Program has a broader view of explosives SSST testing than just performing Proficiency testing. The overall effort is to collect and disseminate high quality SSST test data of HMEs. The first step is to engage a small group of participants to set criteria for testing and methods to produce data acceptable for comparison. For this, three DOE and 2 DoD laboratories were chosen as participants. This has been the work of the Proficiency Test. Ultimately, the group will be broadened to include the international community. The IDCA will also incorporate data obtained from sources outside the group of participants. In addition, the participants will determine if SSST test protocols need to be modified for handling HMEs vs. standard explosives. The group will also share and distribute SSST testing data to the larger explosives community. Ultimately these tests results will reside in a Compendium with controlled access, available to legitimate researchers internationally.

2 CONTINUATION OF THE IDCA PROGRAM FOR FY 2011

The Proficiency Test experimental effort will be completed with FY 2010 funds, and the test reports will be issued comparing each of the materials as measured by the participating laboratories. A final report will be issued summarizing the findings of the Proficiency Test. The IDCA plan for the future is to utilize the results of the Proficiency Test and communicate the results to the HME community in general. Methods to utilize these results are delineated below.

2.1 Restructure the SSST Compendium

Early last year, a beta version of the SSST Compendium was issued to the sponsor. This version was limited in its scope to include testing details on only a couple of categories of materials. It was also crudely formatted to include core data, and speculate on additional safety testing data that might be of use. The test data came from LANL and LLNL, mostly through the NEXESS program. Much of the data had been vetted through the NEXESS program and by the FBI, but was not formally compared to other sources. It was collected in a form that was not optimal for communicating the data. The methodology was largely independent from each laboratory and was not described in any detail. In addition, the sources of the materials studied were not controlled, nor even specified.

The restructuring of this reference will include making it user friendly for access to the most important SSST testing data, descriptions of what methods were used to take the data, standard and reference data, and extra techniques to assess sensitivity of a material that might not be assessed appropriately by standard techniques. An IT person will be employed to assist with this effort.

2.2 Population of the SSST Compendium

With the Proficiency Test completed at the end of the FY 2010 cycle, the SSST Compendium can now be revised in form and populated with vetted, comparable data. The sources for the test data will start with the results of the Proficiency Test. Additional supporting data from the participating laboratories will also be considered for the same and related materials. Recent data from the NEXESS and the IDD programs will also be included when the Compendium has been formatted for web-based distribution.

2.3 Reacquiring Proficiency Test Data with Modified Methods

The premise of the Proficiency Test is to minimize differences in materials and procedures; so individual laboratory performance could be compared on each material. As will be shown below, several gaps in the IDCA analytical methods were realized as the Proficiency Test progressed. Some of these issues were discovered after the specific material had been tested and the reporting completed. For example, the KC/Sugar data had been completed long before the change to different grit size sandpaper for drop hammer had been agreed upon. As a result, the different participants acquired the KC/Sugar drop hammer data using different grit sandpaper. The IDCA team would like to go back and reacquire data on selected materials to update the analysis.

3 PRIORITIZATION OF FUTURE TOPICS OF INTEREST TO THE IDCA

During FY 2010 efforts to conduct the Proficiency Test, many gaps in knowledge about SSST testing appeared when attempting to resolve differences in results among the participants. Some of these gaps are simply issues that face SSST Testing in general and some are new analysis issues associated with HMEs. Particular issues that have arisen are 1) differences in equipment among the participants, 2) differences in methods among the participants, 3) differences in environmental conditions among the participants, and 4) differences in materials handling issue due to the HME formulations. These issues have led to the list of research topics below. These topics are proposed to solve some of the problems encountered as well as anticipated during the Proficiency Test.

3.1 Participation in the International Round Robin SSST testing.

Part of the IDCA charter is to provide SSST testing information to the International Explosives Community in the form of the Small Scale Safety Thermal Testing Compendium. The IDCA is involved in the International Round Robin (through TSWG). The involvement on this level is just through interactions and method comparisons on paper, but the International Round Robin is planning an experimental testing comparison of two standardized materials next year. The IDCA would like to be experimentally involved, at least some of the participating laboratories. The plan is to test and scale-up (multiple tests) two standards that will be provided from one source. The results will be then used to set the baselines for comparing US and European test results. This round robin testing is critical because of the differences in methodologies for SSST testing between the US and the International Community. Typically, the European community uses UN listed methodology, while the IDCA uses DOE and DoD protocols. These are substantially different such that round robin testing of a few standards, at least, are necessary to adequately compare results.

3.2 Developing a fast screening method by DSC for thermal analysis (relate to ARC, ODTX, APTAC, isothermal DSC)

In this last year, several tests performed by LLNL, SNL, NSWC-IHD, NEXESS, the FBI, and for the IDD project have shown that many HMEs are not thermally stable. Currently, thermal stability for testing these materials has been a fairly long process using large quantities of materials. Because this is very time consuming and generally exposes researchers to more risk, the ability to predict this instability in a SSST-type test would be extremely helpful. For this, the IDCA would like to adapt a DSC or other routine testing equipment, like a TGA, to be able to determine thermal excursion (reaction run away) kinetics to sufficient accuracy that will be usable in scale-up processes. This will allow for a quick and easy determination, using a very small quantity of material of whether a mixture has a sufficiently long stability window, at a specific sample size, that it can be handled safely, or will the material have to be handled completely. The IDCA will bring into the screening method, a scientific approach based on thermally characterizing the material with several methods that have kinetic models associated with them (such as ARC, ODTX, APTAC, etc.) These experiments and models will ultimately be used to design an isothermal DSC technique. DSC was chosen because it is ubiquitous in explosives safety testing, while these specific techniques are not.

3.3 Impact of aging of solid-solid, liquid-liquid and solid-liquid mixtures on testing sensitivity.

The IDCA shares information on preparation of samples as one of the ways to assure that every participant is doing the same thing. This is in the effort to be able to compare equipment and method performance and to remove variables such as sample preparations and component sources. A recent issue came about when preparing a solid-liquid mixture for the Proficiency test. Initial results from three of the participating laboratories were completely different. After some discussion, the culprit cause the issue was tentatively decided to be the volatility of the liquid component and that there were sufficient differences in times from mixing to measuring as well as handling of the mixture among each of the participants doing the testing. This simple issue has been resolved. However, there are many more like these in HMEs that are more complicated and need to be addressed. The three principal time-dependent issues (aging) are:

- chemical reactions causing physical and chemical changes in the materials (oxidation-reduction)
- evaporation of liquid component causing changes in formulation
- slow adsorption of water of at least one component causing changes in solubility of the other component.

These issues all cause potential changes to the results of SSST testing if the sample preparation times and storage are not strictly controlled. These issues can also cause the SSST testing results to be inaccurate (causing potential safety issues). The IDCA would like to study some of the more critical examples to establish criteria for mixing times and sample stability.

3.4 Expansion of camera approach to SSST testing

In SSST testing, many of the tests have vaguely defined positive indications. For several of the tests, such as drop hammer, friction and ESD, the “go” is a pop or flash, or physical evidence of smoke. For a given test, however, these indicators are subjective to each participating laboratory, as well as to each operator. Confusion comes when there is marginal reactivity that is hard to separate from the operational noise of the equipment. For example, drop hammer has substantial background noise from the dropping weight that can interfere with a marginal positive detection. For this test, in the IDCA alone, there are differences in declaring a positive reaction. LLNL and LANL use microphones for recording positive events in impact testing and a positive is a certain decibel signal. The other participants use operator-determined positives. There is a need to be more quantitative and more instrumentally based to make these determinations. SNL has been applying optical methods in detecting positive ESD determinations. These results also demonstrate that it is possible to use an instrument for determining a positive event in ESD. The IDCA would like to continue to this line of improving detection in SSST testing:

- Correlating these new techniques with results from current, more subjective methods,
- Expand the list of materials tested to understand the broader range of material response, including the different sound and sights these may cause
- Apply optical and sound techniques to friction and impact

- Investigate other supplemental techniques that may assist in determining positive events.

The ultimate aim of this effort is to remove the impact of the operator in the determination of a Go-No go event. The inclusion of HMEs into the SSST testing arena has caused a much larger variation in output. This would greatly assist the field in more standardization of results and potentially less scatter in intra and inter laboratory testing.

3.5 Effects of impure source materials on testing

In the IDCA, there has been strict control of the materials used for testing—all from the same batch for all participants. The decision to do this was based on the need to eliminate component variation due to different sources. It avoids having to deal with variations due to purity of the components. In FY10, this has been critical for comparing results from the Proficiency test. However, in the real world of testing, the origins of the components and respective purities are not controlled, nor, in many cases, are even known. For FY11, it is now important to answer some of the questions that would be more likely to be encountered in every-day testing—what is the impact of trace impurities on SSST testing performance and results. Because HMEs are often mixtures, these impurities could cause chemical as well as physical changes to the sample. The IDCA would like to study the impact that purity, for example a solid oxidizer from different manufacture and/or process, will have on SSST Testing results. The results of this effort will be a better understanding of the sources of variations in SSST testing of HMEs when comparing results from different laboratories testing the same HME materials.

3.6 Effects of porosity of solid-solid and solid-liquid mixtures on sensitivity

A key issue in SSST testing of HME mixtures is the ability, on a very small scale, to adequately hand mix heterogeneous materials, such as solid-solid and solid-liquid mixtures. This has been best demonstrated by recent work in the IDD program that has shown for selected solid-solid mixtures that the friction sensitivity changes upon scale-up. That difference is most likely due to the change in the mixing equipment—hand mixing in very small quantities to roller mill mixing at larger quantities—but it illustrates how difficult it is to get a representative sample. IDCA generally only mixes very small quantities of materials for testing, as well a most other facilities involved in SSST testing of unknowns. A critical parameter in affording representative testing results is the ability of evenly mixing, followed by random, representative sampling. The IDCA is particularly interested in studying how porosity the affects on mixing and sampling:

- For solids, how does the porosity and particle size effect the way the materials mix and pack together
- For solids, how does the porosity affect the interaction of the solid with the testing apparatus—crushing in impact, stickiness in friction, and transmission of spark
- For liquids, what is the impact of the liquid absorbing into the porosity of the solid
- For liquids, when does the material become thixotropic and flow under testing conditions

The results of this effort will be a better understanding of more sources of variation in SSST testing of HMEs when comparing results from different laboratories testing the same HME materials. In these cases the preparation or synthesis of the material may drive the effects.

3.7 Optimizing sandpaper for impact testing (design of experiments)

The need for sandpaper standardization in the drop hammer test became apparent in the IDCA testing of the RDX standard and then the KC/sugar mixture. The DH_{50} results from three of the 4 testing participants were close, but not identical. Because the equipment used for the testing is roughly the same at each of the participants, the focus of the cause of the differences was shifted to tooling of the equipment. The obvious source could be the sandpaper used for holding the sample. In response, LANL ran two different grit size sandpapers (larger the grit size, the smaller the grit). The results are shown in the Table.

Lab[1]	Sample[2]	Test Date	T, °C	RH, %[3]	DH_{50}, cm[4]	σ, log unit
LANL 150	Powder	12/24/09	20	17	24.0	0.06
LANL 150	Powder	12/24/09	20	17	24.4	0.06
LANL 150	Powder	12/24/09	20	17	23.7	0.05
LANL 150	Powder	4/8/10	24.2	<10	26.7	0.09
LANL 180	Powder	4/8/10	24.2	<10	20.4	0.07

1. LANL 150 = 150-grit sandpaper used in test, LANL 180 = 180-grit sandpaper used in test; 2. form of sample for the test; 3. relative humidity; 4. Neyer method, load for 50% reaction (DH_{50})

The questions that the IDCA are interested in answering are: 1) what is the magnitude of the effect of different grit size on the impact data, 2) what is the role of the binder of the grit, 3) is grit size more important for solid-solid mixtures, such as with HMEs, than for single component materials, such as military explosives. The results of this effort are to empirically determine standard methods for comparison of data. This is critical when reporting data in the Compendium, where issues such as sand paper grit size are critical to know for proper interpretation and utilization of the data.

3.8 Developing methods so ABL vs. BAM data from different methods can be compared

The IDCA is measuring friction by two different methods—ABL and BAM. These methods are being used, BAM primarily in the European community and BAM and ABL in the US community. Both methods use completely different sources of energy for testing—ABL uses pressure driven actuators, and BAM uses weight and gravity driven actuators. The Proficiency Test is using both techniques on a wide variety of types of materials but a limited set of actual formulations. The Proficiency Test will allow for the comparison of ABL and BAM data on identical materials, creating a core database. However, IDCA Proficiency Test examines a limited number of HMEs, and that more testing is needed to facilitate translation between the two friction tests. To compare the two techniques, more has to be done in the more fundamental aspects of SSST testing. This can be met one of two ways—to increase the data base with more empirical data, or determine, on a theoretical basis, a way to translate the ABL data to BAM data and the reverse. The IDCA would like to approach the issue from the latter, by understanding the mechanisms by which each system works, and develop a large statistical set on some standard materials. The end result will be two fold: 1) the ability to use either BAM or ABL friction data to adequately describe safety issues with HMEs, and 2) the ability for the EU and US to exchange and utilize friction sensitivity data.

3.9 Additional HME threats that challenge SSST Testing

HMEs are often formed by mixing oxidizer and fuel precursor materials. Typically, the solid-solid, liquid-liquid, or solid-liquid mixture precursors are combined shortly before use. For solid-solid mixtures, the challenges associated with producing a standardized inter-laboratory sample primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—and taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

The IDCA has chosen several formulations to test that present these challenges. The list below shows what has been addressed and what still remains to be done.

HME mixture properties done already:

- Solid-solid of -100 mesh
- Solid-solid of natural particle size
- Solid-liquid with a volatile non dissolving liquid
- Solid-solid with energetic fuel
- Solid-solid with metal
- Liquid-solid with reactive materials (paste or slurry)

Challenges not addressed

- Solid-solid with very large particle and very small particle
- Solid-liquid with the solid is soluble in the liquid, but there is not much liquid
- Bigger range of solid oxidizers with similar fuels in the Proficiency test

ACRONYMS AND INITIALISMS

AFRL	Air Force Research Laboratory
AN	ammonium nitrate
D	detonation velocity
D-BREIE	Data-Base of Range Evaluated Improvised Explosives
EGDN	ethylene glycol dinitrate
HME	homemade explosives
HMX	
HP/F	hydrogen peroxide/fuel
IDCA	Integrated Data Collection Analysis
IHD	Indian Head
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
ME	mechanical energy
MEKP	methyl ethyl ketone peroxide
P	detonation pressure
PETN	
SNL	Sandia National Laboratories
SO/F	solid oxidizer/fuel
SSST	small-scale safety testing
TATP	triacetone triperoxide
TSA	Transportation Security Administration
UN	urea nitrate
US/EU	United States/European Union

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